

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Lescoche Philippe Examiner: Mellon David C.
Serial No.: 10/532,560 Group Art Unit: 1797
Filed: April 25, 2005 Docket No.:
Title: Membrane for tangential filtration and production method thereof

DECLARATION OF GRANGEON André PURSUANT TO 37 C.F.R § 1.132

I, the undersigned, GRANGEON André, of French nationality, doctor of university of Lyon I (France) in the speciality of catalyse, past chairman of the board of TAMI Industries, with an experience of 25 years in the field of ceramic membranes, inventor and co-inventor of 15 patents in this field.

I am familiar with the English language and I am familiar with the patent application identified above, and with the prior art Childs et al. (US 7,247,370), Garcera et al. (US 6,375,014), Grangeon et al. (US 6,499,606) and Pirbazari et al. (US 5,505,841) discussed in the prosecution of the above-mentioned patent application. I am specifically aware of the contents of the outstanding Final rejection in this case, dated September 14, 2010.

I would like to complete my declaration dated November 18, 2009 especially in order to comment the Examiner's positions in his final rejection.

I. Point 6 of his Final rejection, the examiner considers that the man skill in the art would change the position of the pore-filled layer of Garcera et al., making a number of factual findings, without pointing to anything in the prior art that supports this findings. The advantages alleged by the examiner are not obvious for the man skilled in the art, as the moving of the pore-filled zone could not have been considered to have all these advantages.

First, the examiner mentions: "By having the pore filling facing the separation layer and not opposite to it, the benefit of reduced pressure drop across the membrane would be recognized." It cannot be the case as the only pressure considered by the man skilled in the art is the difference of pressure between the inlet and the outlet of the separation layer, that is to say the pressure inside the channels reduced by the pressure inside the support porosity. And this difference of pressure is, of course, dependent of the presence of a pressure break (the pore-filling zone) but is independent of the position of this pressure break inside the support and as a result independent of its proximity to the separation layer.

Secondly, the examiner considers that: "By moving the pore filling to the facing side, there would be the elimination of the potential of having a fluid stagnation zone". When in fact, by moving the pressure break closed to the separation layer, the potential of having a fluid stagnation zone is the same as the porosity volume crossed by the fluid is the same. The contact time of the fluid inside the porosity of the support is not dependent on the position of the pressure break (the pore filling zone) but is dependent on the porosity and the geometry of this support. As a result, by moving the pore filling to the facing side, the potential of having a fluid stagnation zone is the same.

Thirdly, the examiner follows: "In the event the separation layers were to fail, one of skill in the art would recognize that by having the pore filling immediately following the separation layer, the benefit of reduced fouling would be immediately recognized." This is no truer than the other two assertions made by the Examiner and discussed above. Indeed, most of the time when a separation layer fails to work it is because this separation layer is plugged and the presence of the pore filling zone closed to the separation layer will not improve the filtration. In other cases, when the separation layer presents a defect corresponding to a porosity not as reduced as desired, the presence of the pore filling zone closed to the separation layer would no more allow the required filtration because as shown in the example of Garcera et al, the porosity of the pore filling zone is most of the time larger to the porosity of the

separation layer. So, the filtration cannot be achieved by the pore filling zone when the separation layer fails.

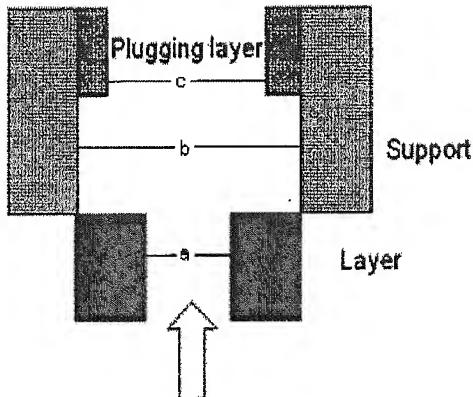
In my opinion as one of skill in the art in the field of the invention and the cited prior art, the Examiner is justifying the obviousness rejection with advantages that one of skill in the art would not find accurate. Because of this, the reasoning used to justify the modification of Garcera et al. is not proper and this means that the rejection is also not proper.

II. In his report dated the 14th of September 2010, the examiner indicates in paragraph 4 beginning on page 6, "While the pore filing of Garcera et al is not explicitly recognized as providing a separating effect, it would inherently provide a size exclusion based separation." The examiner also writes on page 7: "Inherently, a reduced pore size provides a separation effect by size exclusion type separation. In other words, particles in a fluid that are larger than the reduced pore size would not pass through the pore filing simply because they do not fit."

These statements are in total contradiction with the teaching of Garcera et al. Indeed, in Garcera et al, the surface of the channels arranged in the support is covered with one or more layers of filtration (col.1 lines 9-11) that ensure the filtration. The so-called membrane is the assembly of a macroporous support and such a filtration layer (col.1 lines 14-15).

Before reaching the reduced pore size area (pore filling zone) in the periphery of the support, the fluid passes through the filtration layer and the macroporous support (col. 5 lines 28-33).

The figure below schematically represents a membrane according to Garcera et al. It is made of a filtration layer with pore size (a), deposited on the internal surface of the support, support with pore size (b), and of a reduced pore size (c) closed to the external surface of the support. The figure cannot represent the actual relationship between the diameters (a), (b), and (c), since the ratio between (c) and (a) can be higher than 40 as shown in the examples discussed below. The arrow represents the fluid to be treated.



This liquid is generally composed of particles and liquid suspension, of molecules and liquid solvent, this last liquid being possibly the same as the liquid suspension. The aim of the filtration layer is to stop the particles (in the case of a microfiltration operation) and/or the molecules (in the case of ultrafiltration operation) where the steric hindrance is such that it does not allow the passage through the pores. As far as all the molecules or particles have not all been stopped, the liquid that has passed the filtration layer is composed of particles and/or molecules which steric hindrance is lower than the pores they have crossed.

The aim of the support media is to support the filtration layer and to ensure the strength of the whole. Its hydraulic resistance must be extremely low in order not to slow the liquid that has passed through the filtration layer.

The aim of the reduced pore area closed to the external surface of the support is to slow the liquid that has passed through the filtration layer in order to reduce its flux. This reduction is decreasing from the inlet to the outlet of the tubular membrane to compensate the effect of transmembrane pressure drop due to the circulation of the fluid. This speed reduction is obtained by the partial clogging of a variable thickness of the external area of the support. In this area, the porous volume of the support is reduced, which reduces both the porosity and the pore diameter.

The following table summarises the characteristics of porosity and pore diameter of the support, the clogging layer and the filtration layer in the example 6 from the Garcera et al.

example number in Garcera	Layer (a)		Support (b)		Plugging layer (c)	
	Pores diameter (µm)	Porosity (%)	Pores diameter (µm)	Porosity (%)	Pores diameter (µm)	Porosity (%)
1			12	33	4	8
2	0.1		12	33	4	8
3	3		15	45	10,5	12
4	3		15	45	7.5	20
5	0.45		4	15	0.4	4
6			8	30	1	4

Examples 1, 3, 4, 5 and 6 show the variation of the characteristics of porosity and pore diameter of the support before and after the plugging. In all cases we observe in the plugging area a strong reduction in porosity, which is normal since this operation is to clog the pores and thereby to reduce the porous volume. It is the same for the diameter of pores which necessarily reduces. However, reductions in porosity and pore diameter do not seem dependent.

In the examples 2 to 5, the membrane is complete because it is composed of the layer, the support and the plugging area. The fluid to treat that has passed through the filtration layer with pore size 0.1 µm is composed necessarily of particles or molecules which steric hindrance allow the passage through 0.1 µm pores, and so all the more through pores 120 times bigger (case of the support with pore size of 12 µm) or the pores 40 times bigger (case of the clogging area where the pore diameter is 4 µm). It is therefore obvious that the clogging area cannot stop molecules or particles from the filtrated liquid. It is the case in all examples cited in Garcera et al where the filtration layer has a pore diameter similar, or as in the major cases much lower than those of the plugging area.

So there is no molecule or particle stop by the plugging area, as the latter have already been stopped by the filtration layer. The risk of accumulation of material was not obvious in the configuration proposed by Garcera et al. The only possibility for it to appear for a stay of molecules or particles consists in changing the fluid in the porosity of the support by the action of chemicals such as cleaning reagents.

This phenomenon occurs only in certain very special cases, not mentioned in Garcera et al. This is especially the case of microfiltration of milk, where the porosity of the support includes protein, eg casein. During the cleaning operation in alkali, and soda concentrations $> 0,5$ g/l associated with a temperature of 80°C, it can be observed the formation of very large aggregates that can then be arrested by plugging layer. It will be very difficult to subsequently destroy these proteins insolubilized and aggregated, even with an increased concentration of ash. In this case, the membrane must be changed, but this risk exists even in the case of a support, not plugged in the periphery as in Garcera et al.

For the reasons given above in this Section II, it is my opinion as one skilled in the art that separation effect alluded to by the Examiner as inherently occurring in Garcera et al. is not an inherent one and that the Examiner has drawn this conclusion without a proper factual basis. The representation shown above for Garcera et al. reveals that plugging area of Garcera et al. does not produce a particle or molecule stop, i.e., a separation effect.

III. Critical to the examiner's reasoning for making the rejection is the reliance on the placement of the gel impregnation layer in Childs et al. as being on either the feed side of the planar membrane or the side opposite thereto. I contend that it is error for the Examiner to use the teaching of Childs et al. in this regard to modify Garcera et al. More particularly, I maintain that the impregnation with a gel used in Childs et al cannot be compared to the impregnation used in Garcera et al. because there is at least three (3) fundamental differences:

- their function is different: in Garcera et al. the impregnation is used as a pressure brake and cannot ensure filtration. In Childs et al. the impregnation is used as an integrated filtration layer,
- their nature is different: the gel used by Childs et al. is porous in itself. The gel increase the flux whereas in Garcera et al. the material used for impregnation is not porous and provides a partial cogging of the support in order to brake pressure. Even the organic material (epoxy resin polymer such as PTFE)

cited column 6 lines 8-11 of Garcera et al. cannot be compared with the hydrogel or polyelectrolyte hydrogel used by Childs et al.

- their localization is different: in Garcera et al., the impregnation is at the periphery of the multi-channel support and there is a porosity gradient in the direction of flow of the fluid to be treated whereas in Childs et al. the density of gel impregnation is greater or adjacent to one major surface of the planar membrane.

Furthermore, since Childs et al. clearly teaches the use of a pore filled region of the support with a cross linked gel as filtration layer, even if a man skilled in the art would like to use this teaching on Garcera et al. he would simply change the separation mean and replace the separation layer deposited on the support used in Garcera by the pore filled region taught by Childs et al. This pore filled region with a cross-linked gel has to be very specific in order to achieve its separation function and is fundamentally different from the partial clogging with inorganic particulates proposed by the claimed invention which acts as a pressure brake. Since the gel impregnation used in Childs et al. is not even similar to the pressure brake of Garcera et al., one of skill in the art would not use the varying placement of the gel impregnation in Childs et al. to say that it would be obvious to change the position of the pressure brake in Garcera et al.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 04 March 2014

By: A. Garcia

